IS 744 (2000): Textiles - Methods for Determination of Wool Fibre Diameter, Percentage of Medullated Fibres and Kemp Fibre (Amalgamating IS 1348 and 2899) [TXD 1: Physical Methods of Tests]

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IS 744 : 2000
(Amalgamating IS 1348 : 1971
and IS 2899 : 1965)

(Reaffirmed 2006)

भारतीय मानक

वस्त्रादि — ऊन रेशे का व्यास, मैड्यूलेटिड रेशों तथा कैम्प फाइबर का प्रतिशत ज्ञात करने की पद्धति

(तीसरा पुनरीक्षण)

Indian Standard

TEXTILES — METHODS FOR DETERMINATION OF WOOL FIBRE DIAMETER, PERCENTAGE OF MEDULLATED FIBRES AND KEMP FIBRE

(Third Revision)

ICS 59.060.10

BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 4

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Physical Methods of Test Sectional Committee had been approved by the Textile Division Council.

This standard was first formulated in 1955 and later revised in 1966 and 1977. Based on the experience of Wool Research Association, Thane it is being revised again and this revision amalgamates IS 1348 : 1971 'Method for determination of kemp content of raw wool' and IS 2899 : 1965 'Method for determination of percentage of medullated fibre in wool'.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

TEXTILES — METHODS FOR DETERMINATION OF WOOL FIBRE DIAMETER, PERCENTAGE OF MEDULLATED FIBRES AND KEMP FIBRE

(Third Revision)

1 SCOPE

1.1 This standard prescribes the procedure for determination of diameter of wool fibres in any form by means of a projection microscope. It also prescribes a method for determining the percentage of medullated fibres and kemp fibre content of raw wool.

NOTE — In the case of dyed, bleached or finished fibres, the diameter as determined, may be different from that of the same fibres not subjected to such treatments. Further, the estimates of fibre diameter of the same lot of wool at the various stages of processing may not necessarily be the same.

2 REFERENCES

2.1 The Indian Standards given below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of standards given below :

IS No.	*Title*
5910 : 1977	Fineness grades of wool (*first revision*)
5911 : 1977	Fineness grade of wool tops (*first revision*)
6359 : 1971	Method for conditioning of textiles

3 TERMINOLOGY

For the purpose of this standard, the following definitions shall apply.

3.1 Medulla

Hollow or nearly hollow tubular space found within the cortical layer of wool fibre.

NOTE — Medulla may be either dot medulla or ordinary medulla. In the former, the medulla occurs as dots whereas in the latter, its width varies from a very thin streak to more than half the width of the fibre.

3.2 Non-medullated Fibre

The wool fibre without medulla.

3.3 Partially Medullated Fibre (Hetro)

Discontinuous medulla.

3.4 Hairy Fibres (Coarsely Medullated Fibres)

The medullated fibres whose medulla is continuous.

3.5 Kemp

Kemp is highly medullated wool fibre that is the wool fibre whose medulla has width of the fibre or 80 percent of the total diameter of the fibre. A fibre shed from follicle. It is short, wavy, tapering towards the tip, dead white or opaque which do not take dye when dyed.

4 PRINCIPLE

4.1 Profile images of short pieces of the fibres are projected on to a screen and the diameter of these magnified images is measured by means of a graduated scale.

5 ATMOSPHERIC CONDITIONS FOR CONDITIONING AND TESTING

5.1 Prior to the preparation of the slide (sample) (*see* **8.2**), the cut fibres shall be conditioned to moisture equilibrium in standard atmosphere at 65 ± 2 percent relative humidity and 27 ± 2 °C temperature (*see also* IS 6359).

5.1.1 When the cut fibres have been left in such an atmosphere for at least 4 h in such a way as to expose, as far as possible, all portions of the fibres to the atmosphere, they shall be deemed to have reached moisture equilibrium.

5.2 The test shall be carried out in standard atmosphere (*see* **5.1**).

6 APPARATUS

6.1 Projection Microscope

It shall comprise a light source, a light condenser, a stage which supports the slide carrying the fibres, an objective, an ocular, a circular screen and satisfying the following requirements :

a) The stage is movable in two directions at right angles by means of a sliding mechanism capable of successive displacements in 0.5 mm steps.

b) The objective and ocular are capable of providing 500X magnification.

c) The screen with graduated scale is able to rotate about its centre in its plane.

If this screen is not transparent, it shall carry a transparent scale, 5 cm wide, graduated in millimetres along its underside, movable diametrically across the screen between guides.

If the screen is transparent, the transparent scale, graduated in millimetres and used to measure the width of the projected image, shall be placed along one of the diameters. The graduated scale shall be able to rotate about the centre of the circular screen and in its plane.

In the centre of the circular screen, there is a circle whose diameter is equal to a quarter of the optical distance between the ocular and the centre of the screen. All measurements shall be made inside this circle.

d) The magnification of the projection microscope will be checked periodically by means of calibration slide given with instrument.

NOTE — Since magnification of 500X is used during the measurement of fibre diameter, the observed values in millimetres can be converted into microns, by multiplying them by two (micron = 0.001 mm). Hence, the mean of the observed values, if multiplied by 2, would give the mean fibre diameter in microns.

6.2 Microtome

For cutting the fibres to lengths 0.8 mm, 0.6 mm or 0.4 mm.

6.2.1 A suitable microtome, shown in Fig. 1, consists of the following elements :

a) Steel plate with a slot.

b) Steel tongue, fixed to guides which slides along the plate, and adjustable in such a manner that it enters the slot to a predetermined distance.

c) Steel blade pushers, equal in thickness to the width of the microtome slot, each with a stop plate situated at a fixed distance from one of its ends.

A set of three pushers shall be available, the stop plates of which are situated at distances of 0.8 mm, 0.6 mm and 0.4 mm from one of their respective ends.

6.3 Glass Slide

75 mm × 40 mm (approximately) size.

6.4 Cover Glass

50 mm × 35 mm size having a thickness of 0.13 mm to 0.17 mm.

7 MOUNTING MEDIUM

A suitable mounting medium such as cedar wood oil and liquid paraffin having the following properties shall be used:

a) Refractive index between 1.43 and 1.53 at 27°C;

b) Suitable viscosity;

c) Zero water absorption; and

d) No effect on the diameter of the fibre.

8 SAMPLING AND PREPARATION OF SPECIMENS

8.1 Samples shall be drawn so as to be the representative of the lot. Samples drawn in accordance with the procedure laid down in the specification of the material or as agreed to between the buyer and the seller, shall be taken as representative of the lot.

8.2 Preparation of the Test Specimens

8.2.1 *Raw Wool*

Divide the mass of the samples into roughly 40 zones and take a handful of fibres from each zone. Divide each handful into two (taking care to avoid breaking of the fibres) and reject one-half, choosing the half to be rejected at random. If the fibres are parallel, make the division into two longitudinally, that is, in a direction which avoids selection of fibres by their ends. Divide the retained half into two and again reject half at random. Continue in this way until each portion contains about 25 fibres. The reduced sample containing about 1 000 fibres shall constitute the test specimen.

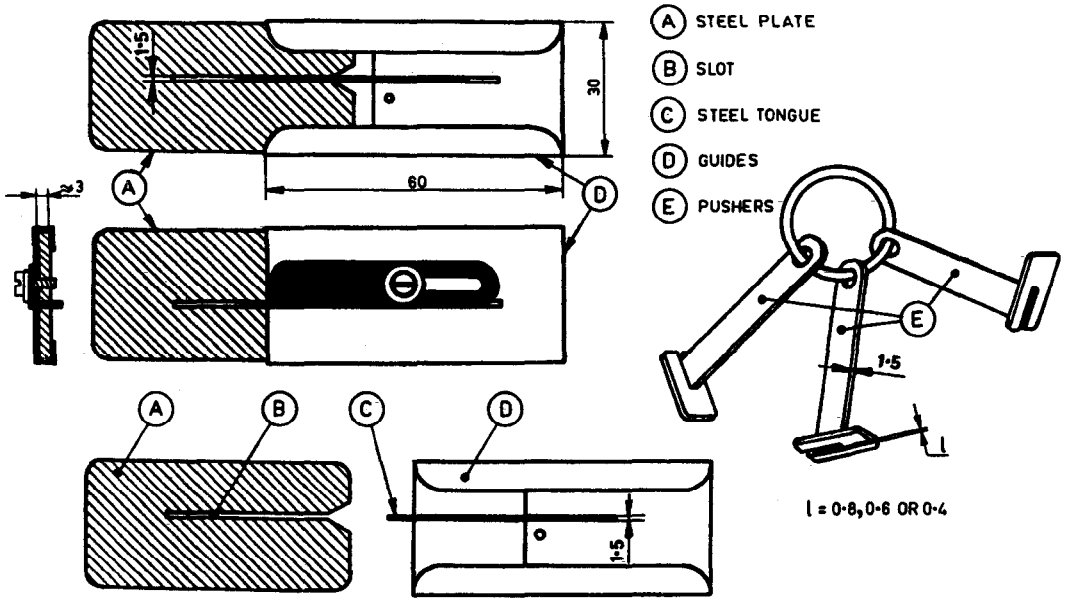
8.2.1.1 Take the test sample as obtained in **8.2.1** and wash it first in benzene or petroleum ether and then in 1 percent solution of sodium oleate at 40°C with two changes of distilled water. Press it gently between two pads of filter paper to extract water and subsequently dry it at a temperature not exceeding 60°C.

8.2.2 *Sliver*

From the bulk sample draw ten pieces of sliver from different portions at random, each of approximately 600 mm length. Take out the small portions of fibres from different places of the ten sliver pieces. Combine these portions of fibres to form a composite sliver of about 600 mm length. This shall constitute the test specimen.

8.2.3 *Top*

Take four sections of sliver each about 1 m in length from different balls of top selected at random. Take only one ball from any one bale or package. Each of the four sections of the sliver shall constitute the test specimen.



All dimensions in millimetres.

FIG. 1 DETAILS OF MICROTOME

8.2.4 *Yarn*

Cut approximately 3 m length of yarn into at least 20 sections, in case of woollen spun yarn or 50 sections in case of worsted spun yarn. Yarn sections thus prepared shall contitute the test specimen.

8.2.5 *Fabric*

Take two samples of at least 50 mm × 50 mm from different portions of the fabric samples which shall represent different warp and weft threads (wales and courses in the case of knitted fabrics). Remove 20 (if woollen spun) or 50 (if worsted spun) warp yarns from each sample. Remove 10 (if woollen spun) and 25 (if worsted spun) weft yarns from each sample. In the case of knitted fabrics, remove 20 threads. The undisturbed pieces of fabric or the teased out yarns of the fabric shall constitute the test specimen.

9 PREPARATION OF SLIDES

9.1 Cutting of Fibres

Take the specimens obtained as in **9.2** and place a representative part of the specimen in the open microtome slot. Then insert the steel tongue and push it strongly to compress the specimen. With a razor blade, cut off the projecting fibres flush with both faces of the steel plate. The cut part of the fibres will then remain in the microtome slot. But forcing the pusher from one side, the cut fibres can be forced out at the other side to a length of 0.8 mm, 0.6 mm or 0.4 mm, according to the pusher used (*see* Table 1).

With a razor blade, cut the emerging fibres flush with the steel plate. Then condition the fibre pieces to moisture equilibrium in standard atmosphere (*see* **5**).

Table 1 Choice of Pushers

(*Clause* 9.1)

Sl No.	Fibre Form	Average Diameter	Size of Pusher (Distance Between Stop Plate and End of Pusher)
		µm	mm
(1)	(2)	(3)	(4)
i)	Raw wool, sliver and roving	> 27	0.8
		< 27	0.4
ii)	Yarns, fabrics	> 27	0.6
		< 27	0.4

9.2 Preparation of Slide

Transfer the conditioned fibre pieces on to a slide and put a few drops of any suitable mounting medium, such as cedar wood oil, to fibre pieces. Disperse the fibre pieces with a dissecting needle into the oil to obtain a uniform distribution. Prepare three slides for each sample. Remove, if necessary, the mixture of fibre pieces and oil to ensure that no oil is squeezed from under the cover glass when it is put on; this would ensure no preferential removal of thin fibres. Put a cover glass by placing one of its edges in contact with the shorter side of the slide and gently lowering the opposite edge. A gentle movement of the dissecting needle on the cover glass will give uniform distribution of fibre pieces in the mounting medium avoiding the formation of air bubbles.

10 PROCEDURE

10.1 Measurement Technique

Mount the slide on the stage. Focus one extreme corner *A* of the Cover glass (*see* Fig. 2) . Move the slide by 0.5 mm in the transverse direction to *B*, then move it by 0.5 mm in the lateral direction. These two movements will bring the first field on the screen.

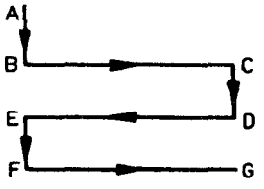
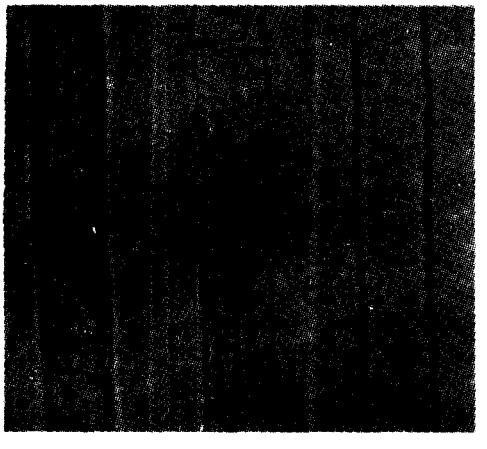


FIG. 2 EXAMINATION OF THE SPECIMEN

NOTE — Generally, both the edges of the image at the point of measurement will not be in focus at the same time since the wool fibres are in general non-circular in cross section. Figure 3 shows changes in appearance of wool fibre as the objective of the microscope is moved away from the fibre. If the objective is too near a fibre edge shows a white border (Becke line). When the objective is too far from the slide, a fibre edge shows a black border (Becke line). When in focus, the fibre edge shows a fine line without a border.

If at the point of measurement, Becke lines (white or black) (Fig. 3A and 3C) appear on both edges of the image. then the objective shall be adjusted so that either the whole image is in focus (Fig. 3B) or one edge of the image is in focus and along the other edge. there is a white Becke line (Fig. 4) .

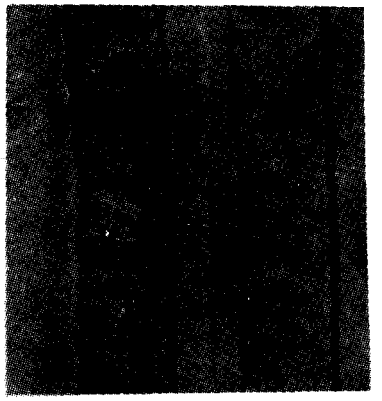
10.2 Rotate the screen until the length of the transparent ruler affixed across it is perpendicular to the fibre image. Move the ruler through its guides until



A B C

A — Objective too near (White becke lines)
B — Fibre in focus (No becke lines)
C — Objective too far away (Black becke lines)

FIG. 3 CHANGE IN APPEARANCE OF WOOL FIBRE AS THE OBJECTIVE IS MOVED AWAY FROM THE FIBRE



CORRECT INCORRECT

FIG. 4 CORRECTLY FOCUSSED FIBRE WITH ONE SHARP EDGE AND WHITE BECKE LINE, AND INCORRECTLY FOCUSSED FIBRE WITH BLACK BECKE LINE

a centimetre division coincides with one edge of the image. Measure the distance between the two edges of the image if the whole of the image is in focus or the distance between one edge of the image and the inside of the white Becke line along the other edge of the image. Record the diameter of that image in Record Sheet (*see* Annex A) .

NOTE — Generally the diameter of the fibre falls between two divisions of the scale. Enter the fibre under the lower whole number of millimetres 'N'. In the subsequent calculation, all fibres recorded under N will be regarded as having a diameter equal to N+0.5 mm.

However, it some times happens that the diameter of a fibre corresponds to a whole number of millimetres 'N'. This fibre belongs at the same time to the 'N-0.5' group (recorded as N-1) and to the 'N+0.5' group (recorded as N). If such a fibre is recorded under 'N-1' group, it is called "underestimated"; if it is entered in 'N' group, it is called "over estimated" . When such fibres measuring an exact number of millimetres occur, underestimate and over-estimate them alternately.

If the sample is to be tested for percentage medullated fibre, then it is also examined for medullation at the point of measurement. Images showing a medulla which does not cross the scale at the point of measurement are recorded as non-medullated. The record shall be maintained after making columns as under and mark the reading in the same way as it is done for finding fibre diameter to calculate percentage of true, hetro, hairy or kemp fibres.

True	*Hetro*	*Hairy*	*Kemp*
No medulla	Broken medulla	Continuous medulla	If the medulla is as big as fibre diameter that is more than 80 percent of the fibre diametre

10.3 Observe the following conventions in measuring the fibre diameter and measurement shall not be made :

— at a point where fibre images cross one another,

— if the extremities of the image do not project beyond the lengthwise edges of the transparent ruler,

— if the length of the image appears to be less than the diameter of the circle on the screen,

— if the image shows the fibre to be longitudinally damaged, and

— if the image has more than half its width outside the circle on the screen.

NOTE — The stage shall remain stationary during the measurement in a given field. It may happen that in a field there will be no fibre at all or only one or two.

10.4 When the fibres have been measured in first field, move the slide 0.5 mm in the lateral direction, thus bringing the second field onto the screen. Measure the fibres in the new field as in **10.2**. Continue in this way along the whole length of the slide. Having reached *C*, on the right hand edge of the cover-glass, move the slide by 0.5 mm in the transverse direction to *D* and continue measuring laterally in 0.5 mm steps and so on. Traverse the whole slide in this way following the path *A,B,C,D,E,F,G*..... (*see* Fig. 2). By following this procedure, the operator has no free choice of the fibres to be measured.

10.5 Repeat the process until at least 200 observations have been made and recorded (*see* Annex A). Calculate the variance (S^2).

10.6 The sample size required to obtain an estimate of mean fibre diameter whithin the desired limit with 95 percent probability level is given by the following formula :

$$n = \frac{t^2 \ S_1^2}{E^2}$$

where

n = number of observations,

t = 1.96,

S_1^2 = variance as calcuated in **9.5**, and

E = desired limit in microns.

NOTE — For value of E, a reference may be made to IS 5910 and IS 5911 for wool and wool tops respectively.

10.7 If the sample size so obtained is greater than 200, prepare another slide(s) to obtain the required number of observations (n) as obtained in **10.6** .

10.8 Calculate the mean arithematic measurement in millimetres; obtain the average diametre of fibres in micrometres (μm) at a magnification of 500X, by multiplying the mean arithmatic measurement by 2. The percent co-efficient of variation V is given by the following formula:

$$V, \textit{percent} = \frac{100\,S}{X}$$

where

S is the standard deviation,

X is the average value of the diametre (*see* Annex A).

11 PERCENTAGE OF MEDULLATED FIBRES

Calculate the number of medullated fibre images as a percentage of total number of images examined.

The standard error *SE* of the percentage of medullated fibre is given by the formula :

$$SE = \sqrt{\frac{m}{n}(100-m)}$$

m = percentage of medullated fibres, and

n = total number of fibres examined.

12 KEMP CONTENT

Calculate the number of kemp fibres as percentage of total fibres by the following formulla:

$$\text{Kemp fibre, percent} = \frac{K \times 100}{W}$$

where

K is the number of kemp fibres , and

W is total number of fibres examined.

13 REPORT

Report shall include the following information:

a) Type, form and condition of fibres tested;

b) Average diameter of the fibres in micrometres, percentage of medullated fibres or percentage of kemp fibres; and

c) Number of observations.

ANNEX A

(*Clauses* 10.2, 10.5 *and* 10.8)

EXAMPLE OF CALCULATION

Diameter mm		*Number of Measurements f*	*Deviation from Assumed Arithmetic Mean] e*	*fe*	*(fe)e*
1					
2					
3					
4	0	1	-6	-6	36
5	00	2	-5	-10	50
6	00000 0000	9	-4	-36	144
7	00000 00000 00000 00000 00000 0	26	-3	-78	234
8	00000 00000 00000 00000 00000 00000 00000 00000 00000 000	48	-2	-96	192
9	00000 00000 00000 00000 00000 00000 00000 00000 0000	49	-1	-49	49
10	00000 00000 00000 00000 00000 00000 00000 00000 00000 00000 00000 000	63	0	0	0
11	00000 00000 00000 00000 00000 00000 00000 00000 000	43	+1	+43	43
12	00000 00000 00000 00000 00	22	+2	+44	88
13	00000 00000 00000 00000 00000 0	26	+3	+78	234
14	00000 00000 00000 000	18	+4	+72	288
15	0000	4	+5	+20	100
16	0000	4	+6	+24	144
17	00000	5	+7	+35	245
18		0	+8	0	0
19	00	2	+9	+18	162
20		0	+10	0	0
21	0	1	+11	+11	121
22					
23					
24					
25					
		323		-275 +345	2130
	TOTAL			=+70	

Calculation of mean diameter in milimetres : $10 + 0.5 + \frac{70}{323} = 10.72$ mm

Actual mean diameter of fibres in micrometres : $10.72 \times 2 = 21.44$ µm

Calculation of the variance of the measurements :

Correction = $\frac{70^2}{323} = 15$

Variance (arithmetic mean of the squares of the deviation) = $\frac{2\,130-15}{323} = 6.55$

Standard deviation of the measurements (in millimetres) = $\sqrt{6.55} = 2.56$ mm

Standard deviation of the measurements (in micrometres) = $2 \times 2.56 = 5.12$ µm

Percentage coefficient of variation = $\frac{5.12 \times 100}{21.44} = 23.9$ percent

95% confidence limits for the mean diameter (in micrometres) = $\pm \frac{1.96 \times 5.12}{\sqrt{323}} = \pm 0.56$ µm

95% confidence limits as percentage of the mean diameter = $\pm \frac{0.56 \times 100}{21.44} = \pm 2.61$ percent

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Review of Indian Standards

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